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STUDY OF THE PURIFICATION OF WATER FROM BIOLOGICAL WASTE

FINAL REPORT January, 1962

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SPONSORED BY NASA,
OFFICE OF LIFE SCIENCES,
UNDER CONTRACT NASW-127
DATED 5 FEBRUARY 1960

PREPARED BY SPECIAL PROJECTS OPERATION

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ABSTRACT

Studies were conducted leading to the optimization of the parameters concerned with a technique for the recovery of potable water from metabolic wastes. The technique begins with the vaporization at a reduced pressure of the volatiles contained in the wastes followed by a high temperature catalytic reaction to destroy the entrained contaminants, and concludes with the condensation and collection of potable water.

The method is unique in that it is a simple single stage process requiring an energy input of 0.644 watts per cc of recovered potable water and has a yield of 90 to 95 percent by weight. Further, no pre- or post-treatment is required of either the raw material or recovered product. Flow rates ranging from 100 ml/hr to 4000 ml/hr have been achieved using properly designed reaction tubes.

Chemical, optical and biological tests and examinations have been conducted on the recovered product. It has been adjudged potable in accordance with the U.S. Public Health Drinking Water Standards. Testing with animals and a human subject has shown no detrimental effects.

Apparatus for space flight, designed on the basis of data accumulated during this program, would weigh approximately six pounds. Thus it would be a desirable sub-system for space flights extending for time periods of greater than ten (10) days.

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I. INTRODUCTION

The recovery of water from man's metabolic waste is vitally important in the contemplated long term manned space flights. Simple calculations show that it is impractical weightwise to store sufficient water for an extended flight. It therefore is imperative to find another source of water which may be treated and rendered suitable for drinking purposes. One obvious source is urine and feces which would ordinarily be considered waste material and present problems in disposal. Several approaches to the solution of recovering potable water from urine and feces have been investigated with varying degrees of success. Fundamentally, the ideal system should be light in weight, compact, uncomplicated, and possess reasonably low energy input. The General Electric Company's Space Sciences Laboratory has evolved such a system.

In February, 1960, the Office of Life Sciences, NASA, Washington, D. C. awarded contract No. NASw-127 to the General Electric Company for the continuation of the technique for the purification of water from biological waste. This technique involves the vacuum vaporization of the volatile materials contained in the waste products, the oxidation of the contaminants contained in these vapors, and finally the condensation of the vapors resulting in potable water.

This final report includes the research conducted on this program for the time period February 5, 1960 to January 15, 1962.

II. PROGRAM OBJECTIVES

The program objectives as outlined in the original contract are as follows:

- 1. Design and test an operating prototype system capable of supplying, from biological wastes, all of the metabolic water required by an animal, and extend the results to include applicability to human beings.
- 2. To accurately determine the point in time (break point) in which the utilization of this regenerative system for water supply becomes more economical weightwise than the pre-storage of water.

III. PREVIOUS WORK

Numerous approaches have been suggested in the technique of water recovery from man's metabolic wastes. These techniques include simple vacuum distillation, lyophilization, ion exchange, and zone refining.

Table I, after Hawk (Ref. 14) lists the composition of a typical normal urine. It may be noted that urine contains about ninety-two percent water. The other eight percent is made up of non-volatile and volatile materials, of which sodium chloride and urea constitute the bulk of the former, and ammonia and phenols, the latter.

Consideration of materials regarded as contaminants indicate that urea and ammonia are foremost and require special treatment. Urea when heated to a temperature of 132°C will form biuret HN(CONH), ammonia and ammonium cyanate NH4OCN. Continued heating will result in the formation of cyclic products cyanuric acid, C3N3 (OH) and ammelid, NH2C3 (OH). Because of this, the heating of urea to these temperature levels must be avoided. Urea, under ordinary temperatures of less than 132°C, will hydrolyze slowly forming additional ammonia and carbon dioxide. Free ammonia is present in urine in small quantities (0.7 grams from Table I) and is of course extremely volatile. Consequently, it becomes important to find a means by which the ammonia contained in the urine is rendered harmless. In order to accomplish this task, the investigations have proceeded in such a fashion as to either treat the raw material prior to distillation or conduct several additional steps after condensation including ion exchange or activated charcoal.

Although these methods have been determined effective from the point of view of obtaining potable water, when consideration is made of the fact that these materials must be prestored in sufficient quantity to continue production of potable water, it is easily seen that the over-all process is penalized by the total weight of the prestored materials.

Although it is generally stated to be feasible, lngram (Ref. 1) presents experimental evidence that about fifty percent of the water recovered from urine by distillation only is suitable for further treatment to make it potable. The first and last twenty-five percent fractions contain too great a concentration of ammonia and volatile organics to be suitable for human consumption. Hawkins (Ref. 2) discussed several distillation techniques including vacuum distillation. However, his conclusions state that probably all methods would require post distillation treatments i. e. filtration, ion exchange, etc. to render the recovered water potable. Hawkins also speculated on other possible techniques. (freezing, electro-osmosis, and ion exchange) and

Composition of a Typical Normal Urine	
Water	Units 1200.0
Solids	60.0
Urea	30.0
Uric Acid	0.7
Hippuric Acid	0.7
Creatinine	1.2
Indican	0.01
Oxalic Acid	0.02
Allantoin	0.04
Amino Acid Nitrogen	0.2
Purine bases	0.01
Phenols	0.2
Chloride as NaCl	12.0
Sodium	4.0
Potassium	2.0
Calcium	0.2
Magnesium	0.15
Sulfur, total, as S	1.0
Inorganic sulfates as S	0.8
Neutral sulfur as S	0.12
Conjugated sulfates as S	0.08
Phosphate as P	1. 1
Ammonia	0.7
4	

concluded that theoretically, it is probable that potable water could be obtained from these methods. He concludes that the best probable method was freezing or, after it has been perfected, electro-osmosis and utilization of solar energy would show the greatest advantage with respect to energy. However, since no experimental information was presented the selection was based on estimated energy and volume requirements only.

More recently, Sendroy and Collison (Ref. 3) published the results of a study program using two distinct approaches on the recycling of potable water from human urine. One was an acid distillation and the other was a lyophilization (freeze-drying) technique. Potable water was successfully recovered from both processes. The distillation included the addition of H_2SO_4 and/or $K_2Cr_2O_7$ to the raw urine, boiling out of approximately 85 percent of the original volume, and then pouring over a column of moistened activated carbon. The freeze-drying technique involved the use of a methylcellosolve - dry ice mixture. Upon vacuum drying, nearly all of the original water content of the frozen material was recovered. This sublimate fluid was then subjected to further treatment with activated charcoal to remove the free ammonia vapor and volatile organic substances before it was adjusted potable.

Wallman and Barnett (Ref. 4) in a study conducted to determine the optimum design of several urine-to-water recovery systems covered phase change processes, solvent extraction, ion exchange, membrane process and integrated systems. The phase change processes considered were air evaporation, vapor compression distillation, freeze sublimation, and freeze crystallization; under membrane processes: electro-dialysis, osmionic process, ultrafiltration, reverse osmosis, thermo-osmosis and electro-osmosis. Other considerations were given to solvent extraction, ion exchange, activated charcoal, decomposition of urea with urease and algae nutrient. As a result of their over-all study, a recommendation was made to use simple distillation for flights of 12 man days and a vapor compression system for longer flights. In the vapor compression process the raw urine is heated, the water is evaporated, and the vapor is fed into a compressor. The vapor is then compressed and condensed to form the product. The heat of condensation is transferred to the evaporator for re-use in the cycle. In both of the above methods, the recovered water is passed through activated charcoal before it is judged potable.

Zeff and Bambenek (Ref. 5) report in their prototype model a vacuum distillation process adapted to space flight yielding acceptable flow rates, however the end product is passed through a filter cartridge containing a mixed bed of ion exchange resin filter pads and charcoal to remove the impurities, odor, and color from the distilled product. Halizone (N.F. Abbott) was also added in some cases for the purpose of reducing the bacterial count.

It is apparent that in all cases thus far presented (including the vacuum distillation) although the flow rates and energy inputs may be acceptable, the initially recovered product is usually contaminated with ammonia and volatile organic compounds which impart a urine-like odor and color to the end product. Lyophilization yields the same product as vacuum distillation, but the inherently slower flow rate makes it even more objectionable. The odor and color may be removed by the use of ion-exchange resins and activated charcoal, but large quantities of resins are required because of the high concentration of salts in the urine. Even more unattractive aspects are the energy requirements and the necessity of regenerating the resin before it can be used again. It becomes obvious from the above descriptions that in all of these techniques, pre- or post-treatments are required before the recovered water may be considered potable.

(中) 1955 11 (A) 11 (B) 11 (B)

Konikoff and Bengson (Ref. 6) have reported on a system in which the need for pre- or post-treatment is eliminated. The technique they describe takes advantage of the low energy requirements of a vacuum distillation and, through the use of a catalytic action, destroys the ammonia, volatile organic material, and any pathogenic matter which may be present.

The destruction of ammonia by oxidation over a platinum or platinumrhodium catalyst is a well-known reaction. (Ref. 7-11). Temperatures for the reaction range from 250°C (Ref. 8) to 1300°C (Ref. 7). Catalyst poisoning by hydrogen adsorption upon the platinum catalyst (Ref. 8) dictates that one use a platinum-10% rhodium catalyst (Ref. 8-9). The latter catalyst is less prone to poisoning and has a higher ammonia conversion yield. Poisoned platinum catalyst may be reactivated by treatment with an oxidizing agent. (Ref. 10). It has been reported that catalyst losses have been estimated to be 0.15 g/ton HNO, manufactured for pure platinum and $0.05g/ton\ HNO_3$ for the platinum-rhodium (Ref. 11). Catalyst activity may also be increased by the use of oxygen in the vapor system (Ref. 12). It is, therefore, reasonable to assume that catalytic action and stability of the platinum-rhodium alloy are suitable for the system planned. In consideration of the estimated loss of catalyst given above, the 10 grams of platinum - 10% rhodium used in the planned system would be totally depleted in the production of 200 tons of HNO,. Thus, in the technique reported by Konikoff and Bengson, the catalyst life would be essentially infinite.

IV. BASIC DESCRIPTION OF THE G. E. WATER RECOVERY SYSTEM

The technique of the G. E. water recovery system is described in the reports of Konikoff and Bengson and Konikoff and Reynolds (Ref. 13). It utilizes to advantage the principles of low energy requirements by using vacuum for the initial change of state and a catalytic reaction zone, thus including an essentially permanent means for the elimination and/or destruction of the ammonia, volatile organic materials, and bacterial matters. The method is not penalized by additional weight requirements needed for a second stand-by unit and a regeneration system, such as required by others using ion exchange resin and activated charcoal for final purification of water. Thus, this process is the only single pass operation.

Figure 1 shows a schematic diagram of the process. The waste materials in the still pot are heated until the vaporization temperature is reached, which is determined by the vacuum drawn on the system. This temperature closely parallels the vapor pressure-temperature curve for pure water.

The vapor from the waste materials pass through a high temperature (approximately 1000°C) catalytic zone which contains a platinum or platinum-10% rhodium catalyst. Through the action of the catalyst and high heat, ammonia and the volatile organic materials are reduced to carbon dioxide, oxides of nitrogen, and water. The heated catalyzed water vapors then pass through a condenser section and finally the condensate is collected at the receiving flask. This final distillate is deemed potable without any further treatment.

SCHEMATIC FLOW DIAGRAM WATER RECOVERY TECHNIQUE

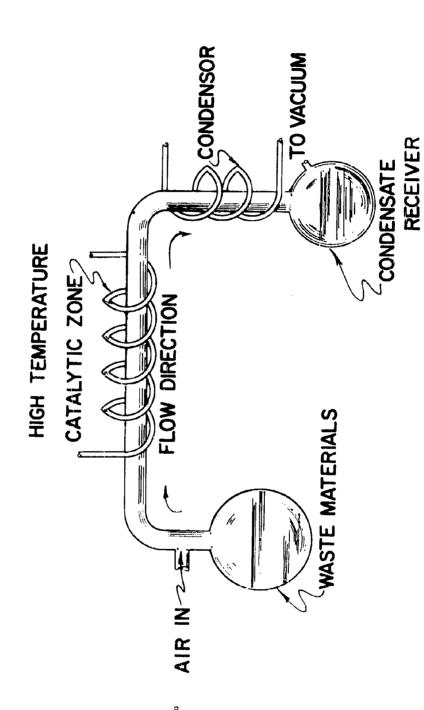


Fig. |

V. RESEARCH AND DEVELOPMENT

Under the study concerned with the optimization of the operating parameters, consideration was given to the following: operating temperatures, heat input, catalyst configuration, flow rates, over-all size and the application of the principle to the utilization of solar energy. Initially, the equipment (Apparatus A) used in the studies was a typical vacuum distillation apparatus including a mullite tube used as the heated catalyst reactor zone. The mullite reactor tube, 70 cm long with glass joint adapters at each end, was packed with Pt-10%Rh catalyst, positioned between the distillation flask and condenser, and was mounted inside a Sentry #2 combustion furnace (Ref. 16). A vapor pre-heater was provided using a Nichrome wire resistance heater. The apparatus is illustrated in Figure 2.

In a later modification (Apparatus B), the apparatus was much lighter and more compact since it was experimentally determined that a vapor preheat was unnecessary before entering the catalytic zone. This simplified the apparatus, reducing the over-all length of the reactor chamber tube in addition to lowering the energy input. Thus a 1¹¹ O. D. McDanels mullite tube, 9¹¹ long, was provided with an externally wound resistance heater made of 17 feet of 20 gauge Kanthal wire imbedded in Norton Alundum cement #1162, and further insulated with granulated asbestos and cement. Standard taper joints were ground on the mullite tube thus eliminating the fragile mullite to glass joint seal. The modification of the furnace reduced the over-all length of the reactor zone by a factor of 4. The present smaller fernace is capable of maintaining a temperature at a maximum of 1350°C (Ref. 16 and 17) and is illustrated in Figure 3.

5. 1 Temperature Studies

In all of the experimental studies presented in this report, one must bear in mind that two different designs of apparatus were used. Essentially, the later variation resulted in a smaller catalyst and vapor pre-heating zone, thus a more compact unit. However, the data obtained from the two units are basically comparable.

Apparatus A was used with a constant volume of urine, relatively constant distillation rate, 10-18 grams of catalyst, and the furnace temperature ranging from 480° - 1250° C. This recorded furnace temperature was comparable to the catalyst temperature. The condensate was collected after processing at various catalyst temperatures and sent to a commercial laboratory for chemical and optical analysis*. In this manner the optimum temperature was determined.

^{*}Betz Laboratories, Philadelphia 24, Pennsylvania

FIGURE 2. ORIGINAL EXPERIMENTAL WATER RECOVERY MODEL - APPARATUS A

FIGURE 3. MODIFIED FURNACE AND WATER RECOVERY MODEL - APPARATUS B

Table 2 shows the composition of potable water following treatment and delivered by the City of Philadelphia, Department of Water (Ref. 15). Table 3 shows a comparison of the analysis of the water obtained using different catalyst temperatures. The higher temperature of 1250°C yields potable water with only 0.1 ppm NH₃ as N, free of odor, a pH of 6.8 and very little total solids as shown by the specific conductance. Figure 4 shows the relationship of total contaminants in ppm as a function of the catalyst temperature.

Water recovered by this technique and using Apparatus A produces a range of pH from 3.9 to 9.1. Experiments using a Pt/Pt-Rh thermocouple imbedded in the catalyst mass were conducted. With flow rate and catalyst mass maintained at a constant, the catalyst temperature was varied and the pH of the resultant recovered water was measured. In Figure 5, the pH of each sample is plotted against the catalyst temperature. The resulting plot indicates that at lower temperatures of 1025°C, a pH of 9.1 was recorded for the recovered water which is close to the borderline of acceptability. At the other extreme, at a temperature of 1115°C, the 3.9 pH was well in the accepted range.

Data obtained from Apparatus B with the smaller heater zone is plotted in Figure 6. Three different masses of catalyst were used, 10 grams, 17.5 grams and 25 grams. In all cases, the catalyst used was packed into one solid mass. With 10 grams of catalyst, the temperature range from 910°C to 1290°C results in a plot which does not decrease below a pH of 9.25 - a completely unsatisfactory water. With 17.5 grams, potable water is obtained at 1180°C; and with 25 grams, a temperature of 1075°C produces acceptable water. Thus, it is seen that with larger quantities of catalyst, the reactor zone may be operated at a much lower temperature to produce potable water. This is universally true; however, no definite conclusion regarding catalyst mass vs temperature can be made without consideration of the catalyst configuration or geometry.

5.2 Addition of Feces

Upon the addition of 75 grams of feces to 1500 ml of urine, a normal water recovery distillation was carried out using 18 gms of Pt screen and a catalyst temperature of 1260° C. The vacuum was held at 60 mm Hg, the pot temperature of the urine and feces mix was 42° C, and an air bleed of 0, 2 CFH. A flow rate of 175 ml/hr, was attained.

Table 4 shows a comparison of the chemical analyses of water obtained from the urine only and the urine and feces. When no heat was used in the catalytic area, the urine and feces mixture produced a foul smelling and highly contaminated product. A comparison of the analysis of the water obtained from urine to that obtained from urine and feces (both using the heated catalytic process) shows essentially the same type of analysis. The total

TABLE 2

City of Philadelphia - Water Department Chemical and Physical Analysis - 1959 Plant Effluent to Distribution (All results in P. P. M. except pH)

Turbidity	Plants on the Schuylkill (Average)
Color	2
Total Solids	243
Total Org. and Amm. (N)	0.18
Nitrite (N)	0.000
Nitrate (N)	1.26
рΗ	6.6
Alkalinity	35
Sulfates (SO ₃)	70
Chloride (C1)	18
Total Hardness	143
Iron (Fe)	0.06
Chloride Resid. Total	0.77
Chloride Resin. Free	0.64

TABLE 3

COMPARISON OF PRODUCTS OF VACUUM DISTILLED URINE

	Raw	distilled	Oxidized in presence of catalys at temperature of:						
	Urine	only	480 ⁰	₽80 ₀	900°	10 15 ⁰	1250°		
NH ₃ as N, ppm	5000	40	20	17.5	8	5.0	0.1		
Total Hardness as CaCO ₃ , ppm	interfr.	2	1	1	1	1	2		
Chloride, ppm	9100	0.5	4.0	0.5	0.5	0.5	0.5		
Н	9.00	9. 9	9. 50	9.80	9. 30	9.45	6.80		
Nitrate as NO, ppm 3	org. in- terfr.	0.05	.05	. 02	5 .025	0.025	0.05		
Nitrite as NO ₂ , ppm	2162	0	+	0	0	0	0		
Color Units	3500	0	2	0	0	0	0		
Phenol, ppm	388, 5	0	0	0	0	0	0		
Odor (intensity)	very str rancid	. str. rancid	str. rancid	str.	str.	faint	none		
Phenolphthalein Alk as CaCO ₃ , ppm	5800	160	48	56	14	14	0		
Methyl Orange Alk. as CaCO ₃ , ppm	20.600	180	80	80	26	24	۷		
Specific Conductance	50,000	72	49	39	27	20	3, 4		

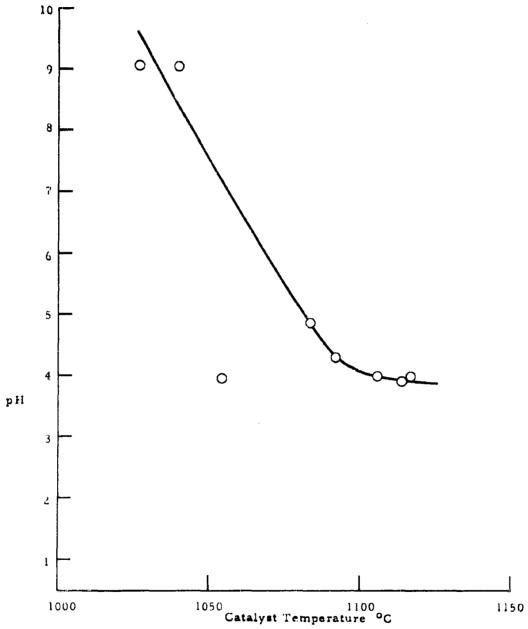


FIGURE 5, Product pH vs Temperature of Catalyst

PH PRODUCT VS. CATALYST TEMP. & MASS

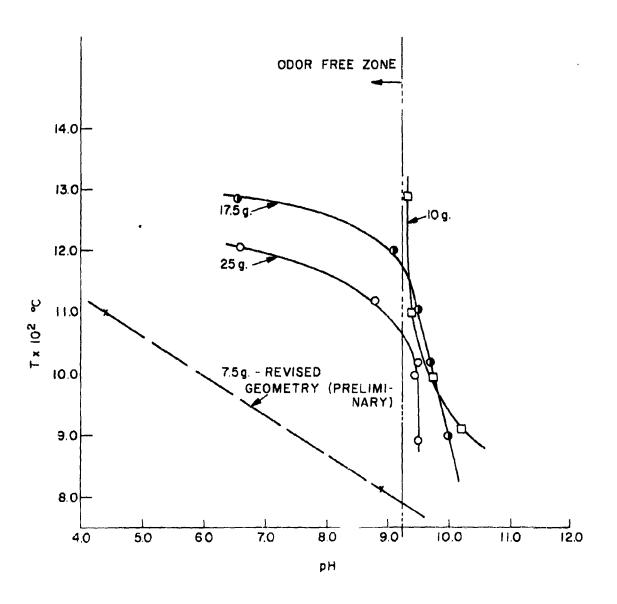


FIGURE 6

TABLE 4

CHEMICAL ANALYSIS OF CONDENSATE TO POTABILITY

	Urine Only fully treated	Urine and Feces fully treated	Urine and Feces (10B) distilled only
Ammonia as N,	0.10	35	1250
Total Hardness	2	0	1
Chloride, ppm	0.5	2	14
рН .	6.8	8,8	10.10
Nitrate as NO ₃ , ppm	0.05	0.025	0.025
Nitrite as NO ₂ , ppm	0	0	138
Color units	0	0	7
Phenol, ppm	0.0	0.υ	3, 25
Odor (intensity)	None	None	Strong Pungent
Total Solids	14	54	168
Phenolphthalein Alk. as CaCO, ppm	0	20	4670
Methyl Orange Alk, as CaCO ₃ , ppm	2	86	5810
Specific Conduct. (micro-ohms)	3.4	180	1400

solids content of the latter is a little greater, but well within the acceptable figure of 500 mg/l (Ref. 23). The coliform organisms, an indication of contamination from fecal matter, is not present, thus showing that the becteria is completely destroyed in the catalytic zone.

5.3 Catalyst Configuration

Surface area is well known to be one of the governing factors in most catalytic reactions. Thus, catalyst geometry would affect the over-all quantity (or mass) of catalyst required. In the experimentation performed on Apparatus A and the initial phases of the work with Apparatus B, the Pt-10% Rh wire screening catalyst was compressed into an oblate spheroid and packed into the reaction tube. The catalyst, when packed in this manner, will present a smaller contact surface area to the vapors. The ideal catalyst will have a structure similar to alumina or silica alumina with a high surface area of the order of one hundred square meters per gram, which is attained by the high degree of internally connected pores. Thus, to achieve this greater surface presentation to the ammonia and water vapors, other catalyst geometries were investigated.

Table 5 lists the types of catalyst configurations used. Initially a platinum filter cone and 3 Pt-10% Rh screens totaling 4 grams were used. The holes in the Pt filter cone retained the vapor flow permitting more vapor-catalyst contact time. With the success achieved using the cone, screens were then packed into several bundles and the bundles separated by ceramic spacers, thus the over-all available catalyst surface area was increased due to the surface presented by the wire mesh. Enough pressure drop is presented by the packed bundles to permit sufficient vapor residence time in the catalyst area to destroy the ammonia and organic compounds. If the same number of screens were used loosely packed, the vapor would pass through at too great a velocity to effect a catalytic decomposition.

To further illustrate the effectiveness of increased surface area, while maintaining the catalyst weight constant, a single spacer equivalent in width to the total spacers used above was placed between two bundles of Pt-10% Rh catalyst. The results of this test showed that although the same mass of catalyst was present, potable water resulted only from the catalyst configuration presenting the greater surface area.

A further series of experiments were conducted using Pt filter pads having an apparently large surface area. These filters consist of tiny identical spheres of platinum welded together into pads, presenting a pore size of approximately 40 microns (!10%). At temperatures above 1150°C and weights greater than 44 g acceptable water is produced. However, the extremely high pressure drop across the pads produced low recovered water flow rates, (60-100 cc/hr.).

TABLE 5
CATALYST CONFIGURATION DATA

Configuration	W eight	Cat. Temp,	pH Recovered H ₂ O	Reference
Cone and Screen	4 g	1200°C	9.0	17
Pt-Rh Screen & Ceramic Spacers	7,5 g	1100°C	4. 4	17
Pt-Rh Screen & Ceramic Spacers	7.5 g	812°C	8} 9	17
Pt-Rh Screen & Ceramic Spacers	7.5 g	1050°C	3,85	18
Pt-Rh Screen & Ceramic Spacers	7.5 g	850°C	8.8	18
Pt-Rh Screen & Single 1/2" barrel ceramic spacer	7.3 g	1020°C	9.6	18
1 - Pt filter pad	22 g	900-1029 (3 runs)	9.6	
2 - Pt filter pads	44 g	1087 (2 runs)	9.6	No previous
2 - Pt filter pads	44 g	1160°C (2 runs)	3,8	reports
3 - Pt filter pads	65 g	1150°C (4 runs)	7.0	

Two specific series of runs were made with Apparatus B, using only 7.5 g of Pt-10%-Rh catalyst and ceramic spacers to improve the over-all catalyst-vapor contact area. The results are plotted on Figure 7 and show a band of T and pH within which potable water is obtained. Comparison of these data with those presented on Figure 6 indicates the great improvement accomplished by increasing the available surface area of the catalyst. A reduction in catalyst weight from 17.5 and 25 g to 7.5 g is achieved and the over-all heat input is reduced proportional to the temperature difference which may be reduced from 1280°C to 930°C at pH = 7.0. Table 6 presents the measured information from 22 runs which comprise the data plotted in Figure 7.

As a result of the above studies, definitive information was acquired as regards the optimization of the catalyst design. Since the energy requirement for the catalytic action is proportional to the mass of catalyst, its specific heat and the temperature differential, it is obvious that the least possible amount of catalyst material will result in the miniumum energy input. These results indicate that circular pieces of Pt-10% Rh 80 mesh gauze, weighing a total of 7.5 to 10 g and separated by two ceramic spacers $3/32^{11}$ wide into three equal parts, will produce potable water when heated to an operating temperature of at least 930° C. The spacer material must be capable of withstanding temperatures somewhat greater than that of the catalyst. Therefore, ceramic materials are recommended,

5.4 Air Flow

For the catalytic destruction of ammonia, the presence of oxygen is known to make the reaction more effective. The present system is based upon the fact that enough oxygen is available in the air over the raw material in the still pot to oxidize all the ammonia and volatile organic matter. Experiments were conducted with Apparatus A to determine the oxygen requirements of the system in order to obtain a suitable product by varying the air flow rates. Initially, the system was flushed completely with argon to eliminate any air present and sealed; then the vacuum distillation was performed. The resulting product from the argon flush possessed an extremely foul odor, thus establishing a need for air in this process. Further experimentation showed no deleterious effects upon the resultant water when the purification process was conducted with air flow rates from 0.05 up to 0.4 CFH.

The same series of experiments were carried out with Apparatus B using the small heater and 17.2 g Pt-Rh catalyst. The results are tabulated in Table 7. Potable water was obtained with 0.006 CFH of air flow (which was the minimum measurable flow). Experimental runs were made for $1/2 \, hr.$, 2 hours, 4 hours, and 7 hours to confirm the results. The odorless, colorless water had a pH range of $7.0^{-\frac{1}{2}}$ 1.0. The variations in flow rates are due to the difference in

pH PRODUCT VS. CATALYST TEMP. CERAMIC SPACERS 3/32" DEEP 7.5g Pt - RH CATALYST

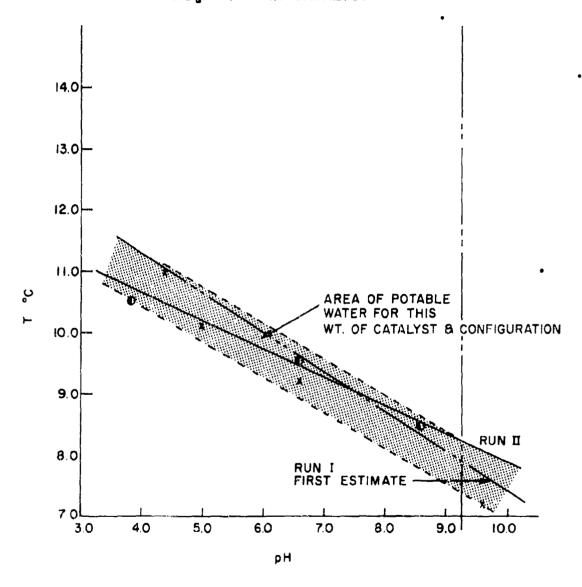


FIGURE 7

FABIE 6

	Odor	none					-	⇒	urine							-	-	-				-	>
Catalyst Configuration Experiments	Hd	0.6	7. 1	4.4	5.0	8.9	9.1	6.6	9.6	4.6	3.7	7. 1	8.1	8.5	8.8	6.7	3.9	3.8	8.0	3.5	7.0	3.4	•
	cc/hr	86	103	83	82	83	86	80	83	80	78	73	80	96	73	92	71	90	96	10	146	99	()
	Cat. Temp. °C	11970	1113	1102	1014	812	899	818	722	961	1057	844	696	366	853	958	1057	1068	866	1102	1121	1091	
	Wt. Ft-Rh	71 *	. pg	~	7.4	7.4	7.4	7.4	7.4	7.4	7.4	7.4	7.4	7.4	7.4	7.4	7.4	7.4	ó.6	ó. ó	6.6	6.6	(
		3 g Pt filter funnel	>	4 porcelain spacers @ 3/32'		•				with a					÷	-							. •
Run	No.		7	~	~]*	Ŋ	9	۲-	က	σ	10		12	13	7	15	16	1.5	18	0 1	20	12	33

TABLE 7

Minimum Air Requirements
17. 2 g Pt-Rh Catalyst

Test	Pot Temp. C	Cat. Temp. ^O G	Air CFH	pH Product	Flow Rate ml/hr.	Actual Exp. Time
1	43	997.5	0.1	7.0	114	1/2 hr.
2	44	966	0.05	8.8	165	1/2 hr.
3	43.5	986	0.03	9. 1	150	1/2 hr.
4	43	1019	.01	8, 5	162	1/2 hr.
5	43	1022	.006	6.7	127	1/2 hr.
6	42, 5	1030	.03	4.7	125	1/2 hr.
7	42, 5	1020	.03	6.1	132	1/2 hr.
8	44	1003	. 03	8.6	192	1/2 hr.
9	43	1000	.05	9. 2	144	1/2 hr.
10	43	1012	.05	9.0	162	1/2 hr.
11	42. 5	1017	.05	8.7	150	1/2 hr.
12	43	1024	.006	6.7	132	1/2 hr.
13	45	1018 •	.006	6.2	105	2 hrs.
14	44	1021	. 006	3. 7	125	4 hrs.
15	44	1003	.006	8, 1	136	7 hrs.
16	43	1000	residual	7.4	64	1/2 hr.
17	44	1007	residual	6, 4	128	1 hr.

energy input in the still. Finally, two experiments were made in which the residual air over the raw urine in the still pot was used as the only source of oxygen. The resultant potable water was clear, colorless, and odorless with a pH of 7.4 and 6.4.

The quantity of air required to process the urinary output of man is an important parameter when consideration is given to the utilization of the vacuum existing in space as the means of reducing the system pressure level so that vaporization of the volatiles will commence. The measured value of 0.006 CFH indicates that with a flow rate of 136 ml/hr approximately 10 hours are required to process one man's daily output (1350 ml total). Thus only 0.060 cu ft. per day will be lost during this process.

5.5 Flow Rate

The permissable flow rate over the catalyst is primarily a function of the catalyst mass and temperature. Thus, the relationship between the vapor conditions and the catalyst is the result of the total heat (above a certain value) necessary to decompose ammonia and the organic materials in the vapor. This was shown experimentally with Apparatus A and 15 gms of Pt-Rh catalyst. At this catalyst mass and a temperature of less than 1000° C, an odorous condensate resulted. However, raising the catalyst temperature to 1100° C resulted in the destruction of ammonia, volatile organics, and permitted the recovery of acceptable water at flow rates from 80 ml/hr to 200 ml/hr. Comparable results were obtained with Apparatus B using sixteen and twenty three grams of catalyst, where flow rates ranged from 92-118 ml/hr (with 16 grams) to a high of 200-215 ml/hr (with 23 grams).

If the basic needs for this process are considered, a certain mass of catalyst and a certain attainable temperature are required. The latter is obviously a function of the heat capacity of the catalyst type and the energy input. Theoretically, if the flow rate was increased by any means available, the heat capacity of the catalyst or energy input must be increased to compensate for the heat transferred to the vapor for initiating the oxidation process which destroys the ammonia, and also to maintain the catalyst at the necessary temperature level.

Unlimited quantities of vapor may, therefore, be satisfactorily processed if one increases the mass of the catalyst as long as the requisite quantity of heat is available. A doubling of the catalyst mass would necessitate a doubling of the energy input due to the increased heat requirements. Similarly, raising the operating temperature could accomplish the same thing, but again energy requirements would be increased. Whereas limits theoretically imposed upon the permissible increase in temperature are due to material restrictions, the catalyst mass may be increased indefinitely as long as an energy source is available to supply the necessary heat capacity of the metallic catalyst.

This has been experimentally demonstrated using Apparatus B where double flow rate (215 ml/hr) was attained at double the catalyst mass and energy input.

5,6 Reactor Heater

Several different means were used, as previously stated, to heat the catalyst zone. In the original apparatus a bulky (160 lbs) Sentry #2 combustion furnace was used. Later, the furnace was modified by using first Nichrome wire and later a small helically wound Kanthal wire resistance heater (20 gauge) around the mullite tube. The completed resistance heater had an over-all dimension of 1¹¹ I.D., 31/2-4¹¹ O.D. and 5¹¹ long. The latter heater has a maximum temperature of 1350°C. In a further attempt to simplify the process, a commercial Kanthal strip-element heater REH-4-30 with a step down transformer was used.

A suitable heater for the reactor section has been evolved using a platinum wire (15-20 gauge) resistance heater wrapped around the mullite tube, imbedded in an insulated with Alundum #1162 cement. This type of heater will withstand extremely high temperatures.

5.7 Self Heating Catalyst

In the search to find improved methods for heating the catalyst, a series of experiments were made using the Pt-Rh screen as both the catalyst and heating source. Initially a 111 wide Pt-10% Rh screen, 17.511 long was used. The ends of the catalyst strips were welded to a length of intertwined strands of Pt-Rh thermocouple wire used as electrical leads. The length of screen was then inserted into the mullite reactor tube and formed into a loose accordian pleat with ceramic spacers. The lead-in wires were brought out through holes in the side of the tube and connected to the step down transformer whose maximum output was 20 volts and 80 amps. In order to increase the available heat, the self heating catalyst unit was further modified by using a longer strip of screen (3611) and by replacing the mullite with a quartz tube (Ref. 20). This extra length of platinum screen provided more contact surface area, higher total resistance (thus more available heat), and a visual observation of the catalyst in order that an optical pyrometer could be used to obtain temperature readings.

Results of the test runs are tabulated in Table 8. The system shows promise in that an acceptable product pH of 8.1 was produced at a flow rate of 400 cc/hr. In this apparatus the catalyst temperature is self regulating since a greater vapor flow rate increases the rate of heat transfer from the catalyst and lowers the temperature of the catalyst, thus lowering the electrical resistance and permitting more current to be utilized. Conversely, if the temperature becomes too great, the resistance increases and thus restricts the current flow.

TABLE 8
Self Heating Catalyst (Pt-10% Rh Screen)

Run	Tube	Cata Length	lyst Wt (gms)	Temp. ^o C	Water pH	Flow ml/hr	(Heater) watt-hr/hr
1	mullite	17.5"	7.3	1100-1200	7.8	172	416
2	mullite	17. 5"	7.3	1050-1150	3, 6	72	493
3	quartz	36.0	16, 5	1025	7. 9	96	495
4	quartz	36.0	16.5	1030	6.0	158	495
5	quartz	36.0	16.5	970	7.6	226	495
6	quartz	36.0	16. 5	1030	4.5	320	600
7	quartz	36.0	16, 5	1000	6.7	225	780
8	quartz	36.0	16.5	1100	8.6	117	775
9	quartz	36, 0	16.5	1030	6.8	304	450
10	quartz	36.0	16.5	1010	8.0	264	450
11	quartz	36. 0	16. 5	1030	8. 1	398	650
12	quartz	36.0	16.5	1030	8, 1	402	650

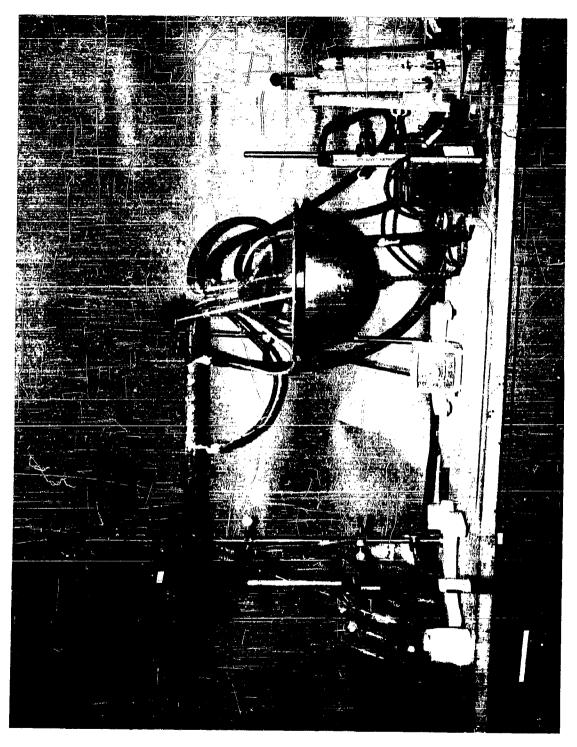
The temperature of the catalyst was approximately 1030°C. The highest flow rate attained was 402 ml/hr, at a pH of 8.1. This required a power input to the heater section of 650 watt hrs/hr. Flow rates were regulated by controlling the heat input to the still pot. Figure 8 is a picture of the apparatus containing the accordian pleated self heating catalyst unit.

Although excellent flow rates were obtained with this unit, the primary concern in its application would be the availability of an electrical source with low voltage and high amperage. This technique could be used very effectively on a space station installation. It may prove quite useful for use with direct solar energy if a sufficiently broad focus of the spectrum can be attained. Obviously, conversion of solar energy to electrical energy is not preferred to direct utilization of solar energy due to its comparative poor conversion efficiency.

5.8 Solar Energy

It has previously been shown (see Section V) that the General Electric water recovery technique may be used in a process where the catalyst alone is heated with no prior vapor pre-heat. As one result of this fact, it becomes apparent that the catalyst heat source may be in the form of thermal energy. Thus experiments were made using a simulated solar furnace, wherein a point source of heat is directed upon the catalyst. The simulated solar furnace was a carbon are image source mechanism placed at the focal point of a 60 inch Army searchlight, Model 1942A. The arc was operated by direct current, furnished from a gasoline-powered Army field generator. The focal length of the searchlight was equal to 26, 26511. Approximately 13 Kw were used to operate the carbon arc. The arc gap at 1-1/4 was maintained constant by an automatic regulating equipment. The radiation from the are anode diverged radially, striking the searchlight parabola, and was reflected in essentially parallel beams along a common optical axis to a second sixty inch parabolic reflector searchlight. The reflected energy was then reconverged by the parabola to form an image of the arc in the focal plane of the mirror on a specially constructed structure to support specimens under investigation.

Temperatures at the focal point of these reflected light rays were controlled by a thermal flux control cylinder. The cylinder was a tubular length of aluminum, mounted coaxially with the optical axis of the mirror and provided with gears for longitudinal movement along the axis. Positioning of the thermal flux control cylinder determined the amount of radiant energy which was permitted to converge on the focal point, thus giving some measure of control of the temperatures available at the sample. Full details and drawings of the arc image furnace may be found in Ref. 21.



To accommodate the reactor tube in the arc image sample holder, a special steel jig was designed to fit in the existing holder. The "inverted π " shaped reactor tube was made from 19 mm quartz tube with glass joints fitted to each leg of the " π " to accommodate the rest of the distillation apparatus. The " π " tube is made with the ends open, packed with 15 grams of Pt-Rh screen, and then sealed off after the catalyst had been tamped hard into position (Fig. 9).

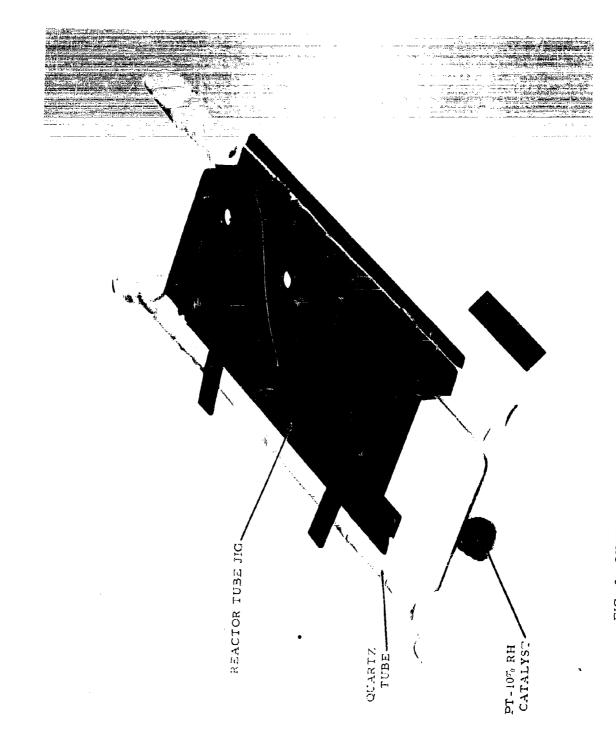
Three successful runs were made with this design. The circumference of the tube was defocused from the focal point by 6 mm in order to heat more catalyst mass with the radiant energy. The heat shield was retracted to a position 6" from the front of the parabola to permit a greater heat flux to reach the catalyst. Figure 10 shows the set-up of the water recovery apparatus adapted to the solar furnace. Figure 11 shows the heat shield reflecting some of the excluded thermal flux as the reactor zone is being heated. Temperatures were recorded optically from a rear peep-hole in the parabola by means of an optical pyrometer. Calibration of the optical pyrometer against a Pt-10% Rh thermo-couple shows a variation of only 5°C, thus verifying the observed catalyst temperature. Table 9 is a tabulation of the data obtained:

TABLE 9
Summary of Data, Solar Furnace Experiments

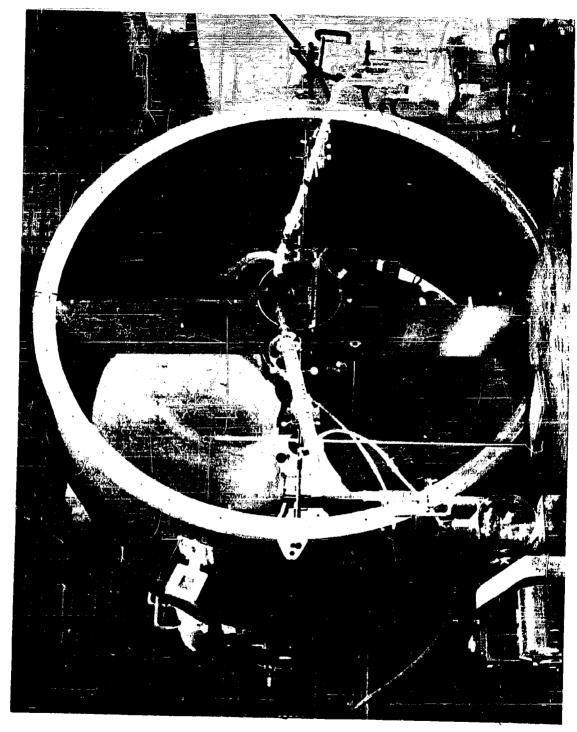
Run No.	Cat. Temp. ⁰ C	Pot Temp. ⁰ C	Flow Rate ml/hr	pH Water	Odor
3	12100	410	150	8.5	none
4	12500	410	150	8.5	none
5	1 2 30 °	38 ⁰	150	ძ. 5	none

The above data clearly indicates that the General Electric water recovery technique is exceptionally compatible with the application of solar energy.

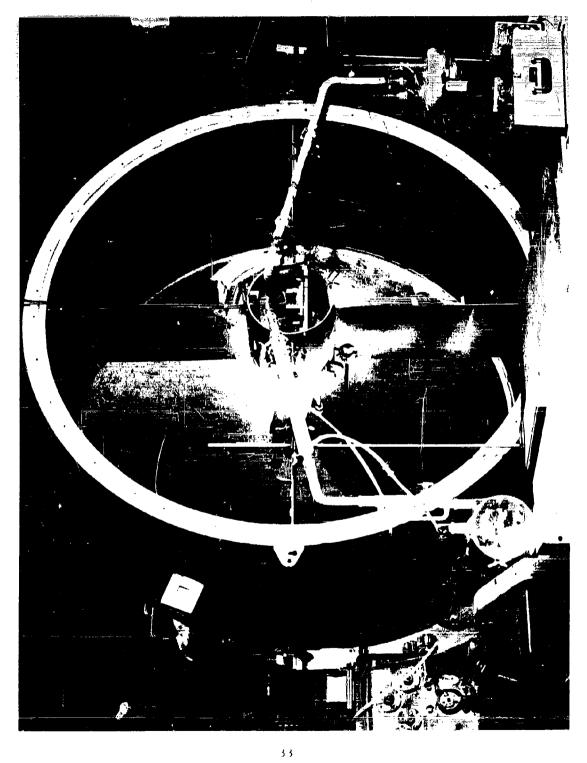
In a space capsule, the sun's rays may be focused with a set of Fresnel lenses upon a mass of catalyst placed in a quartz reactor tube. This eliminates the need for an electrical heat source for the catalyst. Speculating further, space vacuum may be used for the process; and finally, radiant energy from the cabin walls or another set of lenses for focusing the sun's rays could be used as a source of energy to provide the raw material in the still pot with the needed sensible heat and latent heat of vaporization. Thus, virtually all of the power requirements will be satisfied with little or no external electrical source.



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VI. TESTING PROGRAM

A significant number of tests were made to determine potability of the recovered water. The urine samples required for the experimental runs were collected from a mixed group of male volunteers. The operating conditions for the recovery process vary according to the type of apparatus used; however, the general conditions are enumerated as follows:

System Pressure - 60 mm Mercury

Temperature, distillate - 40-46°C

Catalyst temperature approximately 1000-1050°C

Weight of catalyst - 7.5-20 grams

Flow rate = 80-175 ml/hr

Air Bleed - less than 0.2 CFH

The resultant condensate from similar type runs were chemically analyzed by a commercial laboratory as previously stated and were all within the range of potability set forth by the U. S. Public Health Service (Ref. 22). Essentially, all the test runs indicating a pH of 9.0 were deemed potable since at a value of pH 9.25 or greater, slight odor of ammonia was detectable. The U. S. Public Health Service Report (Ref. 23) clearly states that drinking water should contain no impurity which would cause offense to the sense of sight, taste, or smell.

The analysis of the recovered potable water is tabulated in Table 10 and 11 as received from the Betz Analytical Laboratories. These tests show that the potable water conforms with the bacteriological and inorganic requirements of the U. S. Public Health Service Drinking Water Standards. The optical analysis, Figure 12, shows a comparison of the ultra-violet spectrophotometric curves for distilled water, Philadelphia tap water, and recovered potable water. An examination of these plots indicates that the reclaimed water contains insignificant amounts of organic materials. All chemical and spectrophotometric tests show that the recovered potable water from the General Electric process meets the required U. S. Public Health Standards.

6. 1 Animal Studies

A chronic toxicity study was conducted on a rat colony at Bryn Mawr College under the direction of Dr. L. J. Berry (Ref. 25), for the purpose of observing any deleterious effects which might be caused by long term

TABLE 10
CHEMICAL ANALYSIS OF CONDENSATE FOR POTABILITY

Sampling Point		Sampling Point	
Ammonia as N, ppm	0.1	pН	6.80
Carbon Dioxide as CO ₂ , ppm	3	Specific Conductance, micromhos	3.4
Total Hardness as CaCO ₃ , ppm	2	Specific Conductance, micromhos (corrected)	1.7
Calcium as CaCO ₃ , ppm	2	Odor (Intensity)	None
Magnesium as CaCO ₃ , ppm	0	Color Units	0
Phenolphthalein Alkalinity as CaCO ₃ , ppm	0	Nitrate as NO ₃ , ppm	0.05
Methyl Orange Alkalinity as CaCO ₃ , ppm	2	Nitrate as NO ₂ , ppm	0
Sulfate as SO ₄ , ppm	0.4	Total Solids, ppm	0
Chloride as CL, ppm	0.5	Phenol, ppm	0.0

TABLE 11
BIOLOGICAL TEST RESULTS

	Results Period of Incubation			
	24	hrs.	65	hrs.
	20	37	2.0	37
Total Number of Bacteria per ml on Agar	15	25	20	29
Presumptive Test (Coliform Group) Volume of Sample			•	
10.0 ml 1.0 ml 0.1 ml		0/5	•	0/5 0/5 0/5

REMARKS:

This sample as tested conforms with the bacteriological requirements of the U.S. Public Health Service Drinking Water Standards.

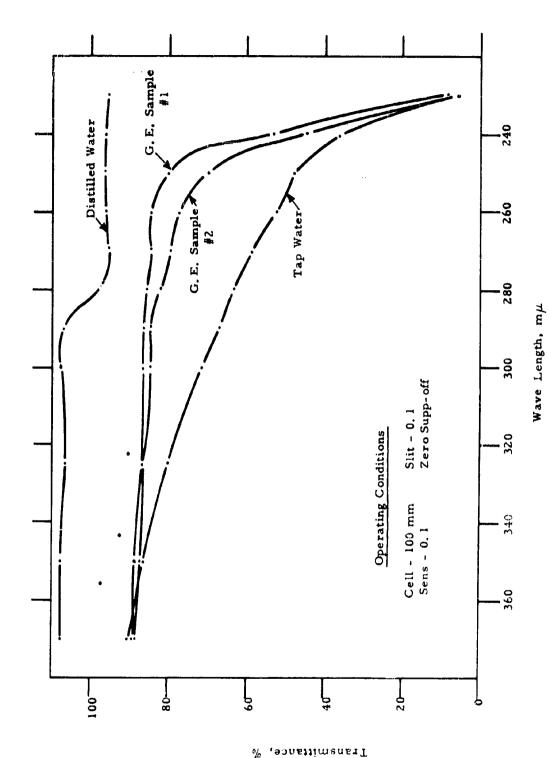


FIGURE 12. U.V. Transmittance (Distilled Water Reference)

feeding with water recovered from urine. A control colony was also maintained as a reference. The experiment was continued for over two generations of rats.

Pregnant rats delivered normal litters on schedule with no difference as to number, size or growth rate. No anomolies were observed in fur color, or size of organs. Appetite, volume of water consumed, and appearance of excrete did not vary from the control group.

In addition to these gross studies, gross histological studies of the organs from the control and experimental animals were made by Bryn Mawr College (Ref. 26). Six organs were examined: adrenals, spleen, liver, intra peritoneal lymph nodes, thymus, and kidneys. All examinations were reported as showing negative results for two generations of rats. The report is reproduced in whole as Appendix A.

The results of these tests provided the impetus to move on to the next test subject, the human being.

6. 2 Toxicity Effects on Humans

Capt. R. A. Bosee, M. C., USN, Director, Air Crew Equipment
Laboratory at the U. S. Naval Air Material Center, Phila., Penna., volunteered
to be the first human to subsist entirely on water recovered by this method
for an extended period of time. To supplement water lost through respiration,
spillage, etc., additional urine was collected at the General Electric Space
Sciences Laboratory, processed, and the recovered water was shipped to
Captain Bosee. Thus, liquid obtained from this process was the sole source
of beverage for this subject for six days. Instant coffee, tea, and fat free
milk were prepared with the potable water as the liquid additive.

Prior to the recycling process, a daily sample of the subject's urine was soutinely analyzed for specific gravity, pH, color, and albumin and sugar. After recycling, the potable water was analyzed for epinephrine, norepinephrine and pH. Since the water from the laboratory model had previously been analyzed for bacterial and urinal content, and had met the Public Health Department's standards, these tests were not conducted during the test.

During test period, the subject's urinary wastes were analyzed daily by standard clinical procedures. The specific gravity varied between 1.011 and 1.023. The latter figure was recorded on the last day of the test. The urine pH varied between 5.0 and 6.0. Urine color varied from light yellow and clear to a yellow and cloudy on the last day. Albumin and sugar analysis were determined and reported negative.

The daily tests conducted on samples of the recovered water were made for pH and retained epinephrine and norepinephrine. The latter two tests were made by using a modified vonEuler method. No traces of either of these amines were present.

The test was concluded on October 23, 1960 after a six day experimental period. Captain Bosee stated that he felt no ill effects. He further stated that "the recovered water was palatable, free of odor, and well received." Ref. 24 contains the complete test protocol and results and is included in part as Appendix B. Thus, it has been demonstrated that the General Electric water recovery process using a catalytical oxidation technique is capable of producing potable water which generates no toxic effects on humans.

VII. DESIGN CONSIDERATIONS

7.1 ACEL Water Recovery Unit

As a logical follow-up of the human toxicity test, a full scale water recovery unit capable of processing 45 quarts per day of wash water and urine, using the subject purification technique was designed and installed at the Air Crew Equipment Laboratory of the Naval Air Material Center, Philadelphia, Pa. in January 1962 (Ref. 27).

This engineering model was installed to the Air Crew Equipment Laboratory Bioastronautic test chamber in which eight day experiments will be made on a six man crew. The system is designed to process the urinary output of six men plus the daily wash water from these men. It was estimated that a 1:4 ratio of urine to wash water would be the composition of the starting raw material resulting in approximately 45 qts. per day to be processed. At a recovery rate of 1 gal. per hour, the total time for processing the water requirements of the test subjects will be 11 1/4 hours. Preliminary results from these planned tests have been received and indicate satisfactory performance. This unit was designed strictly to test the principle of the General Electric water recovery system.

The stainless steel unit is 1911 in diameter and 5811 tall, designed as 3 separate sections; the top heat shield, still pot and reactor zone, and the condensing and collection pot. The reactor section is made of 4 1/4¹¹ O. D. and 3 3/4" I.D. mullite tube over which is wrapped two parallel coils of Pt resistance wire (15 mil.). Figure 13 is a schematic diagram of the water recovery systems. The urine is placed in the still pot and heated by an immers fon heater to about 40°C while the system is being evacuated of air. The vapors follow the dotted path through a set of baffles, picking up some heat from the superheated potable water vapors, pass over the heater wire area, down through the central mullite reactor tube and over the Pt-10% Rh catalyst in the reactor zone. The vapors then impinge upon a set of baffles which direct the steam along the base of the still pot, thus causing a transfer of heat to the original raw supply. Condensation of the vapor is accomplished by copper cooling coils carrying tap water, located in the bottom section of the unit. The condensed water is then collected in the base section and transferred to a reservoir at the completion of the distillation. Figure 14 is a photograph of the unit as delivered. With the operating catalyst temperature at 1000°C, 220 g of Pt-10% Rh catalyst, the pot temperature at 40°C, and the best vacuum available at this vapor pressure, the unit delivers potable water at a rate of 1 gal/hr.

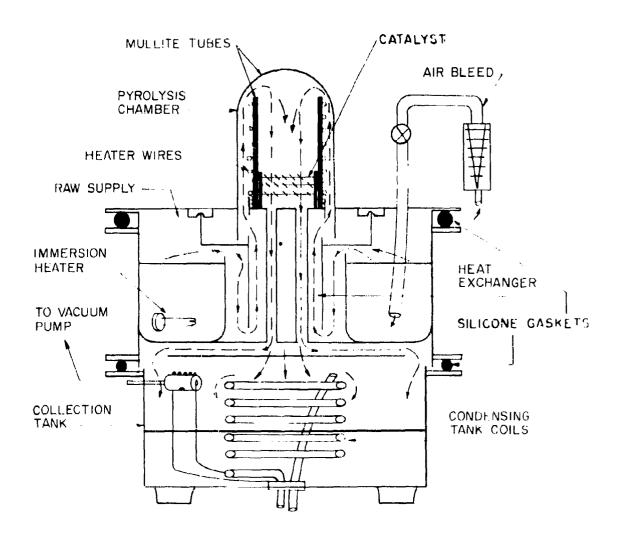


Figure 13 WATER RECOVERY SYSTEM

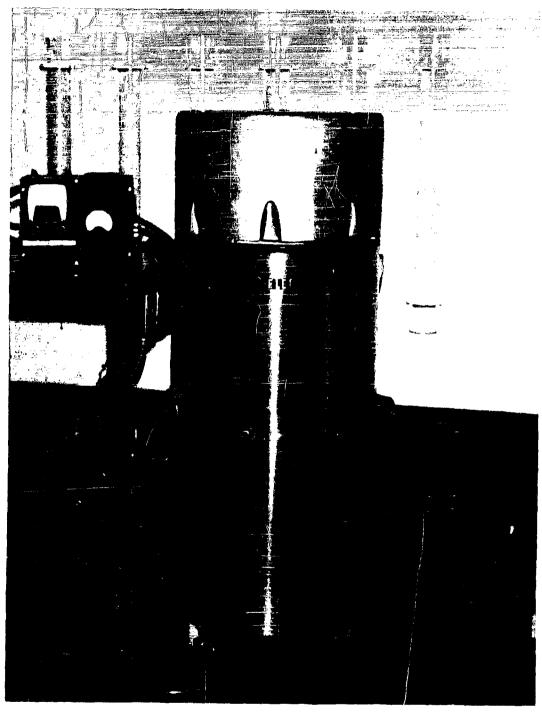


FIGURE 14. ACEL WATER RECOVERY UNIT

7.2 Energy Input

Of great importance in any evaluation of a system for space flight are the energy requirements. Since the General Electric technique for the recovery of potable water from metabolic wastes employs a vaporization, catalystic oxidation, and condensation, the energy requirements may be calculated simply by summation of the energy input for these three steps. In considering the four designs tested in the above studies, the Apparatus A with the Sentry furnace, Apparatus B with the small Kanthal heater, the accordian pleated heater, and the ACEL unit, the sensible heat and latent heat of vaporization will be the same. Thus, with an ambient temperature of 22° C, a still pot (vaporization) temperature of 42° C, and a recovery rate of 90 percent of the total urinary output of man/day (1500 cc x . 90 = 1350 cc) the following heat input is required:

Sensible heat =
$$h_f = wC_p \Delta t$$

= (1350 g) (1) (42-22)
 $h_f = 27,000 \text{ cal.}$

Latent heat of vaporization = $h_{g} = 572 \text{ cal/g for H}_{2}^{O}$

Total Heat input $Q_v = \frac{h}{f} + \frac{h}{f_g} = 799$, 200 cal. or 0.93 Kwh.

At our optimum flow rate of 400 ml/hr a purification run of $1350/400 \approx 3.35$ hours is required to process one man's output per day using the accordian pleated configuration. At this flow rate the measured energy input to the catalytic zone was 450 watts/hr. Thus, the energy input for the heater becomes

$$Q_h = (650 \text{ watts}) (3.35 \text{ hrs}) + 2.2 \text{ Kwh}$$

In this manner, the total heat input, $Q_{\underline{T}}$, becomes

$$Q_{T} = 2,20 \pm 0,93 = 3,3 \text{ kwh.}$$

Further examination of this particular configuration (see Figures 8 and 9) reveals that the apparatus was constructed in such a manner as to permit a large radiant heat loss.

Considering the apparatus in question, the accordian pleated catalyst was placed in a transparent quartz tube and heated by impressing an electric current on it. At the normal operating temperatures (approximately 1000°C) the catalyst glows. Since no insulation was provided, a large quantity of heat was lost to the ambient by radiation through the quartz tube wall.

Remembering that the Stefan-Boltzmann law states that total radiation from a perfect radiator is directly proportional to the fourth power of the absolute temperature:

$$E = \sigma T^4$$

where σ = a constant dependent on emissivity of the material in question at temperature ${}^{0}K$. The emissivity of Platinum at $1200{}^{0}C$ is 0. 167 as obtained from the plotted data, Figure 15, obtained from Ref. 28. The total radiation heat flux then becomes 4.4 watts/sq. cm. Assuming an effective length of 25 cm over which the accordian pleated catalyst is spread:

Area =
$$25 \times 2$$
, $5 = 62$, 5 cm^2 of surface area per side
then $(2 \times 62, 5 \text{ cm}^2) \times (4.4 \text{ watts/cm}^2) = 550 \text{ watts}$

Thus, the actual energy entering into the catalyst reaction is 650 - 550 = 100 watts approximately, the remainder being lost through radiation.

Using this revised value, the heat input to the catalyst now becomes

$$Q_h = (100 \text{ watts}) \times (3.38 \text{ hr}) = 0.676 \text{ kwh}$$

Making $Q = 0.676 + 0.93 = 1.606 \text{ kwh}$

This is equivalent to a heat input of $\frac{1606}{400}$ = 4.0 watts/cc/hr.

As mentioned earlier, an experimental engineering model of the General Electric water recovery apparatus has been constructed for the USN ACEL.

This unit was designed to make maximum use of the heat input so that a more efficient operation could be realized. Test runs made on this amount have resulted in the following data:

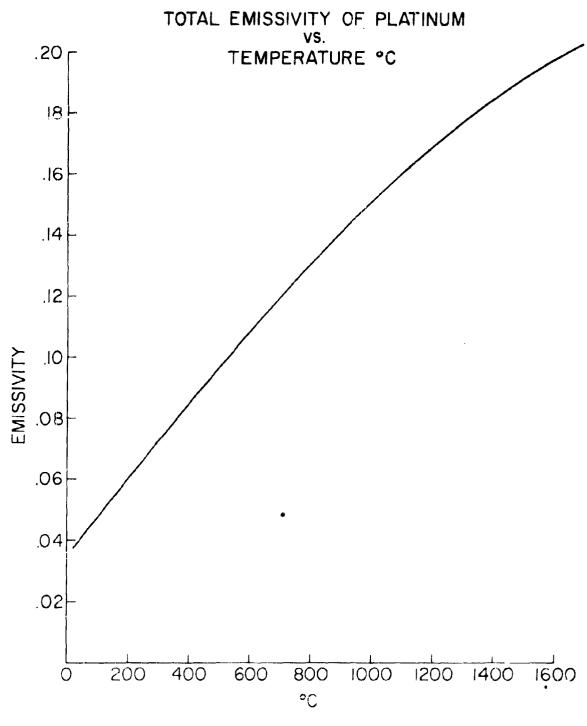


Figure 15

Heat input - heater section 1775 watts Still pot 800 watts

Total 2575 waits

Flow rate = 4000 cc/hr

Thus the heat input is $\frac{2575}{4000}$ = 0.644 watts/cc/hr.

It can now be seen that the application of good engineering design practice can reduce the over-all heat input to a low level. (0.644 watts/cc compared with 4.00 watts/cc).

Table 12 presents the results of these computations.

7.3 Breakpoint

The trade-off point in time when it would be more advantageous to utilize the General Electric water recovery system can now be estimated with a fair degree of reliability. Obviously, from the results presented in Table 12, a unit design which will take full advantage of the catalyst heat input to vaporize the volatiles in the still pot and further to use the heat given up in condensation for the same purpose will be the most efficient. Consequently, using the value of 0.644 watts per cc of recovered product as the energy input for a one man unit, the total energy input becomes 0.644 x 1350 or 868 watts.

Ref. 29 described several methods for the production of energy in space and indicates that values of 150 lbs/kw down to perhaps 5 lb/kw may be expected within the near future. Selecting a fuel cell power supply source, it is reasonable to expect a weight penalty of 25 lbs/kw for this system. Therefore, the power supply system for the water recovery system would be approximately 25 lbs. The hardware itself is estimated to weigh about 6 lbs., thus making a total of 31 lbs.

For comparison, assume a pre-stored water system which will require 5 percent per day penalty for tankage. Thus for one day, the weight carried will be 3.0 ± 0.15 or 3.15 lbs. and for ten days the total will be 31.5 lbs. (we are here only considering the water recovery from urine, hence only 3.0 lbs. per day is considered).

Figure 16 presents this data and illustrates that the breakpoint comes after a time period of ten (10) man days.

TABLE 12

Actual Energy Requirements GE Water Recovery Technique

Operating parameters:		Ambient T - 22° C Vaporization T - 42° C Catalyst T - 1022° C Airflow - 0.006 cfh.		
7 1	Apparatus A	Apparatus B	Accordian Pleat	ACEL
Heat required to vaporize kwh	1040	1040	1040	800(1)
Flow rate, cc/hr	220	1, 50	4.00	4000
Time for process, hrs.	6, 13	0.6	3,38	11-1/4 (based on 45.1/day)
Catalyst heat input watts/hr	1300	9009	650	
Total catalyst heat input watts/hr	0962	5400	2200	1775
Total heat required watts/hr	0006	64, 40	3240	2575
Energy per cc of Recovered H2O	40.9	40, 29	8.10	0.644

Note (1) Additional heat available from condensation of vapor.

BREAK POINT DETERMINATION G.E. REGENERATION vs. STORED WATER SUPPLY

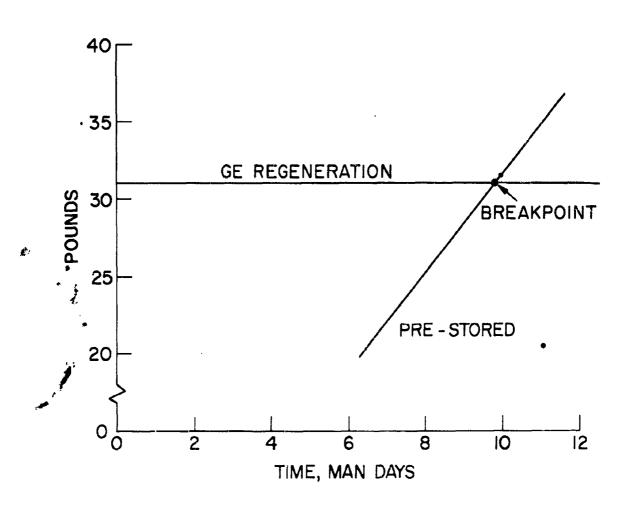


Figure 16

VIII. SYSTEM DESIGN

From the previous studies it is readily seen that the technique for the recovery of potable water from metabolic wastes is feasible and has been demonstrated to be ameanable to an engineering application. Scaling of the unit appears straightforward and is a direct function of flow rate. For capacities greater than one man's output both catalyst quantity, reactor area and heat input must be increased proportionately. The catalyst temperature range remains constant, of course.

Of great importance for the ultimate application of this technique to space flight is its compatability with the space environment, i.e. zero gravity, solar energy, and hard vacuum.

Speculating upon the effects of zero gravity, it appears that the major difficulty will be the separation of the vapor and liquid. This problem is being attacked by several different approaches relating to the utilization of liquid oxygen in space as well as for the utilization of vapor heating/cooling devices. Hence, it appears that a solution will be available shortly. No difficulty is anticipated in the catalyst reaction zone at zero gravity. However, a flight test appears justified.

As has been described earlier, the use of solar energy and space vacuum appear particularly attractive for this system of water recovery. The solar energy (thermal) may be focused upon the catalyst mass by a Fresnel lens system and also upon the still pot. The over-all system may be vented to the "tatmosphere" using proper valving so that the pressure level in the system is reduced to the proper operating value. In this manner, all need for external electrical energy input to the system is eliminated.

9.1 It is concluded that the General Electric vacuum distillation, high temperature oxidation technique for the recovery of potable water from human metabolic wastes is a suitable method yielding a satisfactory product.

The operating conditions which result in the best product at a recovery of 90 percent by weight are, for a one man system:

5 y s	tem pressure	55-60mm Hg *
Vap	orization temperature	42° C
Air	flow, max.	0.006 cfh
Cata	alyst material	Pt-10% Rh - #80 mesh
Cata	alyst mass	10-15 g
Cata	alyst temperature	1000 ₀ C
_ Vap	or flow rate	150-400 cc/hr
Enc	rgy input	0.644 watts/cc recovered product/hr

Operating upon these specifications it is found that the total weight of a one man apparatus (not including the power supply) is approximately six (6) lbs. This weight will increase with the man-day usage. However, the increase will be as a function of the square of the diameter of the reaction zone since the increase is an area relationship.

Biological tests upon one human subject and an animal colony (3 generation of rats) have shown that no detrimental physiological effects are produced from the use of the water recovered from urine by this technique. An experimental full scale model is now under test at the Navy Air Material Center, Philadelphia, Penna.

9.2 In consideration of all the previous data as reported, it is recommended that the study of the General Electric water recovery technique be extended to adapt this system to zero-gravity conditions and to test fly the resultant hardware to prove its capabilities. One of the primary concerns in the zero-gravity flights would be the retention of liquids in a confined area of the still pot, the separation of the liquid

and vapor so that the vapors may undergo catalytic oxidation, and finally the condensation and retention of the condensate at the receiver.

An experimental apparatus should be constructed which will have the capability of operating in a zero-gravity environment, and make use of space vacuum. The latter will aid in vaporizing the volatile materials in urine and serve as the driving force to cause vapor flow from the still pot to the collector.

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APPENDIX A

BRYN MAWR COLLEGE BRYN MAWR, PENNSYLVANIA

DEPARTMENT OF SIGLODY

December 27, 1960

I submit below the final report on subcontract number 214-462136.

The experiments to compare the effects of distilled human urine with tap water as the sole source of fluid intake in rats over at least two generations were carried out according to the protocol agreed upon in advance. This involved the following groups of animals:

1. Female mice bred on arrival at Bryn Mawr College, half of which were placed immediately on tap water and the other half were placed on potable water (distilled human urine). The following litters were born to those ons

A. Tap Water

Date	8/2	14 young	10 female	4 male
	8/4	12 young	9 female	5 male
	8/6	11 young	7 female	4 male
	6/7	13 young	Premale	5 male
	Total	50 young	, 34 female	16 male

One of the mother rats was sacrificed for tissues on 8/51. Two famales and one male from each litter were isolated and mated on 10/24. All others were sacrificed. The weights of the 'emales and males to be bred were followed with the results shown in Figure

B. Potable Water

Date	8/1 8/2 8/4 8/15	12 young 9 young 10 young	5 females 4 females 6 females 4-Zemales	9 males 4 males 5 males 6 males
	Total	39 young	17 females	22 males

One of the mother rats was sacrificed for tissues on 8/51 along with a female from each of the first three litters. A female from the last litter was sacrificed on 10/5 for tissues. Two females and a male from each of the first three litters were isolated and mated on 10/24. The weights of these adult animals are also shown in Figure 17. The animals on potable water were slightly heavier than those on tap water but the parallelism of the growth curves suggests no enhanced growth rate. Perhaps the smaller litter size (a chance phenomenon in all probability) permitted the rats on potable water to grow more rapidly as nurslings.

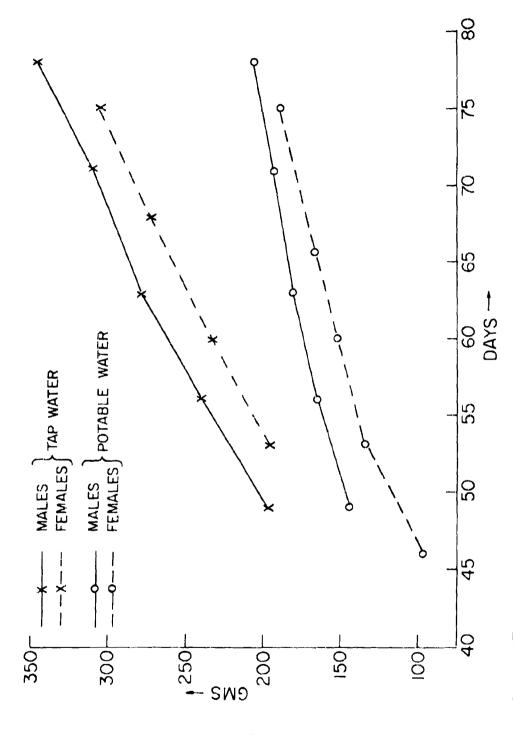


Figure 17

2. Litters were born to seven of the eight female rats maintained on tap water and bred on 10/24 and to all six of the female rats maintained on potable water and bred at the same time. These are shown as follows:

A. T	ap Water			
Date	11/16	8 young	4 females	4 males
	11/16	lū young	5 females	5 males
	11/17	10 young	8 females	2 males
	11/17	15 young	7 females	8 males
	11/18	9 young	5 females	4 males
	11/19	8 young	3 females	5 males
	11/26		6 females	
	11/20	9 young	O TOWNYOR	3 males
	fotal	69 young	38 females	31 males
B. Pot	table Water			
Date	11/17	11 young	5.females	6 males
	11/27	15 young	7 females	6 males
	11/18	10 young	4 females	6 males
	11/18	lő young	9 females	6 males
	11/23	10 young	6 females	4 males
	11/20			
	11/27	10 young	6 females	4 males
	Total	69 young	37 females	32 males

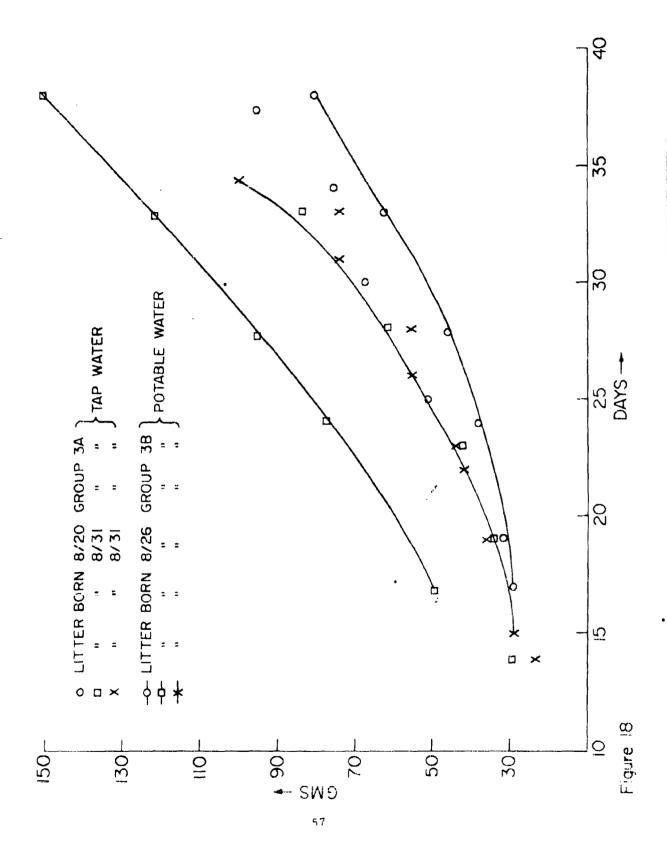
It is of interest to note the close similarity of the two groups. There were 69 offspring born in seven litters of rats on tap water and the same number born in six litters to rats on potable water. The sexes were almost identical. There were 58 females and 31 males on tap water and 37 females and 32 males on potable water.

S. Two groups each of five female rats and five males were mated on 7/25 after the animals arrived at Bryn Mawr College. One group was permitted to drink only tap water and the other group drank only potable water. The results of the matings are as follows:

A. Tap water

Date	8/20	9 young	5 females	4 males
	8/31	7 young	3 females	4 males
	8/31	4 young	2 females	2 males
	Total	20 young	10 females	10 males

One of the mothers was sacrificed for tissues on 10/5. Two females and one male from each litter were mated on 11/7. The rest were sacrificed at the same time. However, animals of both sexes were weighed from the time of weaning until sacrifice with the results shown in Figure 18.



B. Potable Water

Date	8/26	7 young	2 females	5 males
	8 /26	3 young	2 females	1 male
	8 /2 8	8 young	1 female	7 males
	Total	18 voune	5 female	15 males

One female from each litter was sacrificed for tissues on 10/3. The two remaining females plus three from the litter born on 8/13 to an animal in Group 18 were mated with one male from each of the above litters plus a male born on 8/13 to the same animal in Group 18 which contributed the females. The mice were mated on 11/7 and the few remaining males were sacrificed. The growth of these litters is shown in Figure 18.

4. Five of the six females bred in group 3A delivered litters and all five females from group 3B gave birth to young. The results are as follows:

A. Tap Water

Date	11/29 12/1 12/1 12/4 12/4	5 young 9 young 12 young 11 young 10 young	2 females 3 females 7 females 6 females	l male 6 males 5 males 6 males 4 males
•	Total	45 young	23 females	22 males
B. Pote	ble Water			
Date	12/1 12/1 12/2 12/4 12/11	11 young 7 young 11 young 8 young 12 young	4 females 1 females 6 females 5 females 2 females	7 males 6 males 5 males 3 males 10 males
	Total	49 young	18 females	51 males

Summary

The tables that follow give the totals for each group of animals:

A. Tap Water

(a) Bred before arrival at Bryn Mawr College	Number of Litters	Number of Females	Number of Males
(b) Bred at Bryn Mawr			
College	3	10	10
(c) Bred offspring of (a)	7	3 8	28
(a) Bred offspring of (a) (d) Bred offspring of (b)	5	23	22
Total	19	105	76
Average Litter Six	● 9.6		
Females/Males	1.4		

B. Potable Water

(a) Bred before arrival at Bryn Mawr College (5) Bred at Bryn Mawr	Number of Litters	Number of Females	Number of Males 22
College	3	5	15
	6	37	32
(c) Bred offspring of (a) (d) Bred offspring of (b)	<u> 5</u>	18	51
Total	18	77	98
Average litter size	9.7		
Females/males	೦. 8		

The only difference that can be seen at all is the sex ratio in the two groups. On a sample as small as this it is reasonably certain that no significance can be attached to it. A much larger number of offspring would be required before one could say that potable water favors males. This is made evident by the larger difference in sex ratio seen in animals raised on tap water, the fluid universally used routinely in animal rooms. If the sexes on tap water were in equal number then some doubt might be cast on the results with potable water but otherwise not.

The other point that might be made is the greater growth rate seen in one of the litters born to an animal on potable water (Figure 18). This is the litter which grow more rapidly than the others but it contained only three babies. Perhaps each was able to consume more milk and hence grow faster. Since all . litters on petable water showed more rapid weight gain than those on tap water, no great conclusion can be drawn. Actually, where growth of litters is to be compared, it would be scientifically sound if the same total number and the same sex ratio were maintained in each case. This would require some manipulation of litters but in no other way would growth be strictly comparable.

In conclusion, it is the author's opinion that potable water is without demonstrable influence on growth and reproduction in rats over the period of time covered by the experiment.

Respectfully submitted,

Professor of Biology

LJB/vb

APPENDIX B

(Direct reproduction of pages from the Air Crew Equipment Laboratory

Report NAMC-ACFL-459 Sept. 1961)

U. S. NAVAL AIR MATERIAL CENTER Philadelphia, Pennsylvania

AIR CREW EQUIPMENT LABORATORY

PROBLEM ASSIGNMENT NO. CO4AE13-1 ENVIRONMENTAL REQUIREMENTS OF SEALED CABINS FOR SPACE AND ORBITAL FLIGHTS

> Space Age Utilization of Recycled Metabolic Wastes

NAMC-ACEL-459

1 SEPTEMBER 1961

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Director

REPORT NO. NAMC-ACEL-459

SUMMARY

INTRODUCTION

In a consideration of any closed-loop life support system, the problem of obtaining potable water from man's metabolic wastes is one of prime consideration. The technique of producing potable water by vacuum distillation has been advanced by the Missile and Space Vehicle Department (MSVD) of the General Electric Company, Philadelphia. The evaluation by human consumption of the potable water produced by this method constitutes the objective of this report. As such, this report represents a unique effort without precedent in the experimental literature.

SUMMARY OF RESULTS

The analysis of daily urine samples from a test subject revealed normal values in regard to specific gravity and pH. The former determination varied between 1.011 and 1.023; the latter, between 5.0 and 6.0. Qualitative albumin and sugar determinations were negative. Urine color varied from light yellow and clear early in the test period to yellow and cloudy on the last day. Analysis on samples of recovered water revealed excessive acidity on the first three days of the test. The level of pH was maintained at near neutral levels for the remainder of the test. Recycled urine showed no traces of epinephrine and norepinephrine. Daily water intake was maintained, the palatability and odor-free characteristics of the potable water being totally acceptable. The test subject (RAB) suffered no ill effects and the device appears to have good potential for space flight applications.

CONCLUSIONS

From the results of this investigation, it is concluded that drinking potable water produced from metabolic wastes by the vacuum distillation method of the MSVD, General Electric, offers no physiological impairment under the limitations of the test as conducted.

RECOMMENDATIONS

The wide range of uses such water reclamation devices possess calls for further and more elaborate test procedures from the physiological and biochemical points of view. Complete closed loop systems involving several men should be conducted so that a realistic estimate of the operational utility of such potable water devices can be obtained. Careful analysis of the system described in the present report and other systems is needed so that a firm recommendation for space vehicle utilization can be offered. It is recommended that such tests be continued as soon as it is deemed practical.

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METHOD

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Several techniques are currently being investigated for their suitability in recovering water from human waste. Electrodialysis, distillation, filtration absorption with ion exchange, and freeze-drying are but a few of the methods now available. Final considerations and selection of a water recovery system for a space vehicle will depend upon crew size and mission objective. The point at which water recovery systems demand inclusion into vehicular design is reached when the weight of the system is less than that of stored water. Figure 1 presents an approximation of the trade-off values between stored water and water recovery systems. As such it suggests the primary advantage to be obtained from a closed-loop system.

In cooperation with the Missile and Space Vehicle Department of the General Electric Company, Philadelphia, the Air Crew Equipment Laboratory has recently completed a preliminary evaluation of an operating human urinary waste reclamation system. The suitability of the potable water produced by this vacuum distillation device for human consumption was tested on the senior author of the present report, a Naval officer 50 years of age and in excellent health.

Basically, a vacuum distillation process was used with a platinum catalyst employed to remove ammonia and other volatile organic compounds. The apparatus was installed by GE personnel under the direction of Mr. J. J. Konikoff and was capable of a yield of 150-180 ml of potable water per hour. Figure 2 schematically represents the operation of the vacuum distillation device.

In order to complete the tests in as short a period of time as possible, and to allow the water lost through respiration and other metabolic processes to be replaced, the subject's urine was supplemented by urine from other sources. All urines collected were processed; the recovered water was refrigerated, and carried by the subject in conventional thermos jars. The liquid obtained was used by the subject for a six-day period as his sole source of beverage. Instant coffee, tea, and fat-free milk were prepared with the potable water as liquid additive.

Prior to the recycling process a daily sample of the subject's urine was routinely analyzed for specific gravity, pH, color, and qualitative albumin and sugar. In addition, after recycling, the potable water was analyzed for epinephrine and norepinephrine content as well as pH determination. Since the yield of this laboratory's model had been previously analyzed for bacterial content, as well as urinal content, and had met the Public Health Department's standards, these tests were not conducted in the present study.

RESULTS

The subject's urinary wastes were analyzed from day to day in a standard clinical fashion. Similarly, the potable water yield of the distillation device

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was also analyzed on a daily basis so that a constant check on the subject's liquid intake and output could be maintained.

The daily samples of urine which were clinically analyzed presented a normal picture. Specific gravity determinations varied between 1.011 and 1.023, the latter value being recorded on the last day of the run. The ph of the subject's urine varied between 5.0 and 6.0, which was well within the normal range. Urine color varied from light yellow and clear early in the test to yellow and cloudy on the last day. Qualitative albumin and sugar determinations were negative.

Although a complete blood analysis to be performed on a daily basis was originally considered, the approach was abandoned for the following reasons: First, the fact that supplemental urines were used to provide the potable water yield necessary violated the closed-cycle aspect of the study. Secondly, it probably would require very lengthy or stressful treatment to initiate any noticeable or significant change - potable water consumption, in itself, would not create such an effect. A first day sample of blood revealed normal values for leucocytes, erythrocytes, differential count, serum sodium, and potassium.

Two test determinations were performed on samples of recovered water in each daily yield; one for pH, and one for retained epinephrine and nore-pinephrine. The pH determinations did reveal excessive acidity during the first three days of the test: 2.8, 3.2, and 3.2. This was corrected during the remaining days of the run by adjusting the temperature of the catalyst so that the subject's water intake had a pH maintained between 6.0 and 7.0. Tests for the presence of epinephrine and norepinephrine were made using a modified von Euler method (3). The recycled urine showed no traces of either of these catechol amines.

In addition to the quantitative data, let us add that the potable water produced by the device was entirely palatable, free of odor, and well received. Basically, there was little or no significant decrease in the subject's daily water intake. Refrigeration added to the water's acceptability in that it appears to remove the somewhat "flat" taste which is characteristic of all distilled water.

DISCUSSION

To a great extent the data reported here fit in well with earlier work using infra-human subjects. Sendroy and Collison (2) at the Naval Medical Research Institute fed a charcoal treated distillate of human urine to a colony of rats for more than thirty days. Using this potable water as the sole beverage source, Sendroy and Collison failed to find any toxic effects or abnormalities on autopsy. Similarly, a collaborative study between the Space Sciences Laboratory of the General Electric Company and Bryn Mawr College

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has failed to find any ill effects on gestation period, litter size, or growth characteristics in those rats maintained on water recovered from human urine (1).

The present investigation did not indicate any physiological impairment related to consumption of recycled urinary waste. The only change in function at all revealing was the gradual increase in specific gravity of the urine. This might be indicative of some slight decline in water intake and consequent increased concentration of urine solutes. The results we obtained obviously verify previously collected data in that the feasibility of a vacuum distillation process used with a platinum catalyst for providing potable water has been demonstrated. That such a system has real value for space vehicle design can hardly be doubted. System improvements and greater percentage returns will allow such water recovery systems to utilize wash water and air conditioning condensate, as well as urine so that complete closed-cycle metabolic waste converting systems can be built.

Other uses for such systems also present themselves. One very obvious possibility is concerned with utilizing recycled urine and other metabolic waste constituents as an algae nutrient medium. Since algae normally use urea for growth it would seem that human urines - modified in one of many fashions, might be ideally suited for this purpose. Thus, in addition to providing a primary water source, human waste materials can also provide the nutrients necessary for intra-vehicle vegetation growth.

An admittedly speculative but interesting use for such water recovery devices can be seen for long-term occupancy radioactive fallout shelters. These devices, as they become commercially feasible, could be installed in both military installations and civilian protective shelters to obvious advantage. First, in time of radiological or biological attack, it is highly likely that normal water supplies would be contaminated and unuseable. Secondly, storage of dehydrated foods could be made in advance with the knowledge that adequate water would be available.

In short, the possibilities of incorporating water recovery devices extend not only to space vehicles but to other modern day needs. Power requirements, weight and mass considerations, ease of maintenance, and other factors need to be well established before the uses of such devices can become conclusive. The efforts of the past few years have formulated the basic paradigm for all waste recovery devices. This general form is shown in Figure 3. The efforts of the next few years will serve as the distillation process for the notions now current and presented in this report. In the interests of research economy we all hope for a high yield and little waste.

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ABSTRACT

Potable water reclaimed from metabolic wastes by means of the vacuum distillation technique of the Missile and Space Vehicle Department of the General Electric Company was the sole source of water intake in a one-man six day test. Urinary characteristics regarding pH and specific gravity were within normal ranges; qualitative albumin and sugar determinations were negative. pH of recovered water was excessively acidic early in the test period. No traces of epinephrine and norepinephrine were towns in recycled urine. Daily water intake as might be influenced by palatability and odor was maintained constant, the whole of the experimental treatment effecting no embarrassment on the physiological state of the subject.